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FILTER ELEMENTS FROM HIGH-PURITY QUARTZ GLASS FOR FLOW-THROUGH EQUIPMENT FOR CONCENTRATION OF SILICA-CONTAINING FEEDSTOCK

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The results of a study on creation of a porous, permeable material based on molten quartz and technology for manufacturing filter elements from this material are presented. The compositions of the developed materials, process parameters, and physicochemical and filtering properties are reported. The filter elements developed were successfully tested in industrial conditions.

Quartz glass has a special place among all industrial glasses. Having very valuable properties such as high thermal stability and translucence in the ultraviolet, visible, and infrared regions of the spectrum, and chemical resistance to aggressive media (acids, metal melts), it is an irreplaceable material in many cases [1, 2].

The use of silica-containing materials (quartz sand, veined quartz, etc.) for production of quartz glass is rigorously regulated by the composition and amount of impurities in them (maximum of 0.01 wt.%). For this reason, enrichment of silica-containing feedstock is a mandatory operation in preparing it for production of high-purity transparent quartz glass [3, 4]. One stage of this process is chemical concentration of the charge (0.40–0.16 mm particles), which consists of dissolving impurities in acids, washing the concentrate with distilled water, and drying. This is a long operation, conducted in intermittent equipment. Concentration of silica-containing materials in continuous equipment significantly reduces the duration of the process and increases the feedstock quality. The technology and implementation of this process were developed at PL Kontur Co. One of the basic elements of continuous units is the filter element made of high-purity quartz glass, which, as an impermeable barrier for the charge, allows solutions of acids enriched with soluble impurities to freely pass through it. The size of the filter element is: 490 mm in diameter, 40 mm thick.

The basic requirements for a filter element are: permeability of more than $1 \text{ cm}^3/(\text{sec} \cdot \text{cm}^2)$ at pressure greater than 0.5 atm, compressive strength of more than 25 MPa, high wear resistance to the silica charge, chemical resistance to solutions of acids, minimum open porosity of 20%.

Based on an analysis of the scientific and technical literature and patent information, we decided to study filter elements made from large-grain charges of almost single-fraction composition composed of high-purity quartz glass using silica, ethyl silicate, polyorganosiloxane, and other sols as binders.

The initial feedstock for preparing the charge was quartz tubes after chemical treatment manufactured by Zavod ELVAKS State Unitary Enterprise according to TU ShchLO 047-252, with a mass content of 99.99% SiO_2 .

Processing of the paste compositions, technology, and determination of the basic properties of the filter elements were conducted on sample cylinders 60–62 mm in diameter and 23–44 mm high. The samples were formed by the method of semidry, two-sided compression molding in a laboratory hydraulic press at unit pressure of 30 MPa. The charge for formation was prepared in a Z-shaped mixer lined with plastic, with successive addition of the components. The dried samples were annealed in a laboratory furnace with silicon carbide heaters at a temperature of $1250 \pm 20^\circ\text{C}$ with holding for 2 h. The physicochemical properties of the samples after annealing are reported in Table 1.

The microstructure of one of the samples investigated is shown in Fig. 1. The maximum pore size was determined with the “bubble” method according to a method of investigation developed and certified at NTTs Bakor Co. The filtering characteristics of the samples were investigated on a pilot laboratory setup created at PL Kontur Co. The filtration characteristics of the porous, permeable, quartz-glass ceramic are reported in Table 2.

The composition of the materials whose properties best satisfied the client's requirements was selected based on the studies conducted. Samples were prepared for performing full-scale tests (Fig. 2).

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TABLE 1

| Sample | Filler characteristic | | Properties after annealing | | |
|--------|---------------------------|-----------------|----------------------------|------------------|--|
| | dispersion, μm | mass content, % | compressive strength, MPa | open porosity, % | apparent density, g/cm^3 |
| 1–2 | 200–315 | 100.0 | 60 | 36 | 1.60 |
| 3–4 | 315–400 | 100.0 | 55 | 35 | 1.59 |
| 5 | 400–500 | 85.0 | 42 | 26 | 1.82 |
| | 2–4 | 15.0 | | | |
| 6 | 400–500 | 42.5 | 45 | 32 | 1.75 |
| | 200–315 | 42.5 | | | |
| | 2–4 | 15.0 | | | |
| 7 | 500–630 | 85.0 | 40 | 27 | 1.80 |
| | 2–4 | 15.0 | | | |
| 8 | 630–800 | 100.0 | 37 | 27 | 1.80 |
| 9 | 800–1000 | 100.0 | 26 | 26 | 1.82 |
| 10 | 1000–1600 | 100.0 | 20 | 21 | 1.90 |
| 11 | 1000–1600 | 85.0 | 33 | 21 | 1.90 |
| | 2–4 | 15.0 | | | |

* Quartz glass was used as the filler; 1% SiO_2 , with respect to dry matter, was used as the binder.

TABLE 2

| Sample | Height, mm | Permeability,* $\text{cm}^3/(\text{sec} \cdot \text{cm}^2)$, at pressure, atm | | | | Maximum pore size, μm |
|--------|------------|--|------|------|------|----------------------------------|
| | | –0.5 | 0.5 | 1.0 | 2.0 | |
| 1 | 23 | 1.17 | 1.07 | 2.06 | 3.92 | 70 |
| 2 | 44 | 0.79 | 0.58 | 1.28 | 2.62 | 70 |
| 3 | 23 | 1.34 | 1.18 | 2.59 | 4.64 | 130 |
| 4 | 44 | 0.70 | 0.67 | 1.38 | 2.76 | 130 |
| 5 | 44 | 0.44 | 0.36 | 0.80 | 1.27 | 100 |
| 6 | 44 | 0.45 | 0.39 | 0.65 | 1.95 | 120 |
| 7 | 44 | 0.69 | 0.42 | 0.96 | 1.99 | 120 |
| 8 | 43 | 2.06 | 1.74 | 3.31 | 5.49 | 350 |
| 9 | 42 | 2.55 | 2.55 | 3.91 | 6.02 | 400 |
| 10 | 41 | 4.45 | 3.23 | 5.02 | 7.95 | 450 |
| 11 | 43 | 1.78 | 1.46 | 2.58 | 3.53 | 300 |

* The permeability (the rate of mass transfer through the sample) was determined with distilled water.

The tests were performed on a model setup in conditions approximating the real conditions of use of filter elements. The numerical values of the permeability with distilled water were in good agreement with the data obtained on the laboratory samples.

The strength and wear resistance of the full-scale samples were evaluated with the following method: 150 kg of quartz grit were placed in the unit, and 100 liters of water was poured in and drained (the water was pumped at excess pressure of 0.5 atm and 0.5 atm vacuum). The unit was simultaneously tilted from right to left to transfer the quartz grit over the surface of the filter. All of the filter elements passed the test without fracturing.

The acid resistance of the filter elements was evaluated with the following method: previously dried and weighed

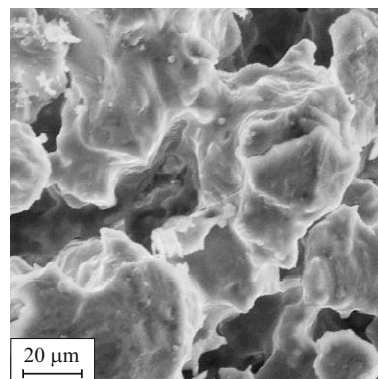


Fig. 1. Microstructure of the porous, permeable, quartz-glass ceramic.



Fig. 2. Full-scale quartz-glass filter element (49 cm in diameter, 4 cm thick).

samples were placed in the working solution of acids (12.6 g/liter HNO_3 + 10.0 g/liter HCl); the temperature of the solution was $22 \pm 2^\circ\text{C}$. After 45 days, the samples had dissolved by 30% maximum. For comparison: samples manufactured by a foreign firm initially proposed for use totally dissolved in this time.

As a result of these studies, a porous, permeable material was thus created from quartz glass and technology was developed for manufacturing them from filter elements which were recommended for industrial use (RF Patent No. 2255792).

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